



10/620,654

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/620,654 : Confirmation No.: 1421  
Applicant : Rabasco, et al  
Filed : July 16, 2003  
For : Polymer Emulsion Coatings For Cellulosic Substrates  
With Improved Barrier Properties  
  
Art Unit : 1773  
Examiner : Tarazano, Donald Lawrence.  
Docket No. : 06326 USA  
Customer No. : 23543

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

<p align="center"><b>CERTIFICATE OF MAILING</b></p> <p>I CERTIFY THAT THIS PAPER (ALONG WITH ANY PAPER REFERRED TO AS BEING ATTACHED OR ENCLOSED) IS BEING DEPOSITED WITH THE UNITED STATES POSTAL SERVICE WITH SUFFICIENT POSTAGE AS FIRST CLASS MAIL IN AN ENVELOPE ADDRESSED TO:</p> <p align="center">COMMISSIONER FOR PATENTS P.O. BOX 1450 ALEXANDRIA, VA 22313-1450</p> <p>ON <u>14 Feb 2006</u> Date Mary E. Bongiorno (Type or print name of person mailing paper) <u>Mary E. Bongiorno</u> Signature of person mailing paper</p>
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**DECLARATION UNDER 37 CFR 1.132**

Dr. John J. Rabasco

The declarant hereby states the following:

1. that he has a Doctorate in the field of Chemistry;
2. that he is employed by the assignee in the above application, i.e., Air Products Polymers, L.P. as a research chemist;
3. that his field of endeavor within the Product Research Group of Air Products Polymers, L.P. resides, *inter alia*, in the development of polymer emulsions for use as pressure sensitive adhesives, laminating adhesives, nonwoven binders, coatings, caulks, grouts, etc.;

4. that as Senior Principal Research Chemist he has considerable experience in the design and development of vinyl acetate based emulsions, including vinyl acetate/ethylene emulsions, for the above recited applications;
5. that he has reviewed the Office Action of October 31, 2005 including more specifically the rejection of Claims 1-18 based upon Worrall, et al US 3,355,322;
6. that at his direction two experimental attempts to reproduce the polymer emulsions of Example #1, Composition B of Worrall US 3,355,322, et al were made and these attempts are identified as Examples 1 and 2 in this declaration;
7. that the experimental attempts were carried out in following equipment and under the following conditions:
  - a) a one-gallon, stainless steel, stirred tank reactor was used instead of a rockable stainless steel reactor;
  - b) the agitation speed was set at 600 rpm;
  - c) the scale was increased by a factor of four to match the reactor fillage as laid out in Example 1 of Worrall, et al;
  - d) Natrosol<sup>®</sup> 250LR was used as the hydroxyethyl cellulose, which is the specified protective colloid for Composition B;
  - e) Disponil<sup>®</sup> A3065 was used as the polyoxyethylene lauryl ether;
  - f) the vinyl acetate and ethylene were charged to the reactor in the weight ratio specified for Composition B, i.e., 58/42 vinyl acetate/ethylene for the initial charge and 68/32 for the vinyl acetate/ethylene feeds during polymerization; and,
  - g) separate feed lines were used to charge the vinyl acetate and ethylene instead of charging as a mixture as specified in Example 1 (the

equipment set up did not allow for charging vinyl acetate and ethylene as a mixture);

#### Example 1

- a) that the reactor was charged with deionized water (958.25 grams), Disponil A3065 (14.15 grams as a 65 wt% active solution of a polyoxyethylene lauryl ether), sodium hydrogen phosphate (12.0 grams), Natrosol 250LR (960 grams, as a 2 wt% active solution of hydroxyethyl cellulose), and potassium persulfate (9.2 grams).
- b) that the above amount of reactants and agents charged to the reactor resulted in an increase over the amounts employed by Worrall, et al by of a factor of four;
- c) that in order to maximize the extent of activity of the thermal initiator, potassium persulfate, and maximize conversion of vinyl acetate and ethylene during the polymerization, the initial monomers were charged to the reactor prior to heating the reaction mixture. (Although Worrall, et al specify heating the above mixture to 70 °C prior to charging the monomers, it was believed better to charge the monomers to the reactor first because potassium persulfate as a thermal initiator begins to degrade and generate radicals when heated to 70 °C. Secondly, Worrall, et al did not specify how long would take to heat the mixture to 70 °C nor did Worrall specify how long the mixture was to be held at 70 °C before charging the monomers. Thirdly, Worrall, et al did not specify how long it took to complete the initial charge of vinyl acetate and ethylene. There is

no disclosure in Worrall, et al that these time periods are critical. Thermally active initiators such as potassium persulfate would have been at least partially degraded by the time the monomers were charged, thus having significant impact on how the polymerization proceeds, i.e., the rate of polymerization);

- d) that 621 grams of vinyl acetate and 450 grams of ethylene were charged to the reactor, followed by heating this entire mixture to 70 °C. This amount of vinyl acetate and ethylene was calculated (at a 58/42 ratio) so as to achieve a reactor pressure of 1800 psig at 70 °C;
- e) that it was found that at 70 °C, the pressure was 2600 psig instead of the specified 1800 psig but within the overall allowed pressure range permitted by Worrall, et al. However, because the reaction was proceeding and the pressure was declining, there was no 1.5h induction period as noted by Worrall, et al. (Worrall, et al provide no disclosure indicating that an induction period is critical.);
- f) that when the pressure declined to 1800 psig the vinyl acetate and ethylene monomers feeds were charged into the reactor at the specified ratio and at a rate to maintain 1800 psig;
- g) that based upon calculations (assuming 100% conversion) sufficient vinyl acetate and ethylene was added to the reactor to provide a final product solids content of 45 weight percent;
- h) that the addition of this calculated amount of vinyl acetate and ethylene was completed within 3 hours (Worrall, et al added the feeds over 6 hours and that may have been due to a lower reaction rate due to the unknown time at which the potassium persulfate was exposed to elevated

temperatures prior to charging the monomers. Again there is no disclosure that introducing a delay feed over 6 hours is critical to producing the product except for the maintaining of reaction rate as opposed to the 3 hour introduction period employed here to maintain reaction rate);

- i) that the final product had a solids content of 39.6 weight percent, 3.6 weight percent unreacted vinyl acetate, and the final product had high levels of grit and coagulum;
- j) that analysis of the final product with differential scanning calorimetry (DSC) showed that the polymer had a glass transition temperature of -25.3 °C and no ethylene crystallinity. There was no melting point transition and no heat of fusion evident for the entire DSC scan which ranged from -65 °C to +200 °C indicating the absence of crystallinity in the polymer. (A copy of the scan is attached hereto and it is appropriately identified ); and,
- k) that the polymer when cast as a film had a tacky surface at room temperature.

#### Example 2

- a) that the procedure of Example 1 was repeated, with the general exception to the procedure for charging the initial amounts of vinyl acetate and ethylene;
- b) that 372.6 grams of vinyl acetate and 270 grams of ethylene were charged to the reactor, followed by heating the mixture to 70 °C. Once at


70 °C, additional vinyl acetate and ethylene was added at the 58/42 ratio until a pressure of 1800 psig was reached;

- c) that once at 1800 psig, it was evident again reaction was taking place, and therefore, there was no induction period. Therefore, the vinyl acetate/ethylene monomer delay feeds were started immediately, as in Example 1 described above;
- d) that calculated amounts of vinyl acetate and ethylene were added to the reactor in a 3 hour time period instead of a 6 hour time period;
- e) that the final product had a solids content of 40.4 weight percent, 5.2 weight percent unreacted vinyl acetate, and the final product had high levels of grit and coagulum;
- f) that analysis of the final product with differential scanning calorimetry (DSC) showed that the polymer had a glass transition temperature of -27.4 °C and no ethylene crystallinity. There was no melting point transition and no heat of fusion evident for the entire DSC scan which ranged from -65 °C to +200 °C indicating the absence of crystallinity in the polymer. (A copy of the scan is attached hereto and it is appropriately identified); and,
- g) that this polymer too, as in Example 1 had a tacky surface when cast as a film at room temperature.

8. that a DSC scan for Example 8 as described in Applicants' specification (and appropriately identified) has been provided as a representative illustration of the differences in thermal properties of polymers having ethylene crystallinity, thermal melting point and

heat of fusion as described in the above-captioned application polymer vis-à-vis polymers of the type produced by Worrall, et al.

That all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements are made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

  
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John Rabasco

Title: Sr. Princ. Research Chemist

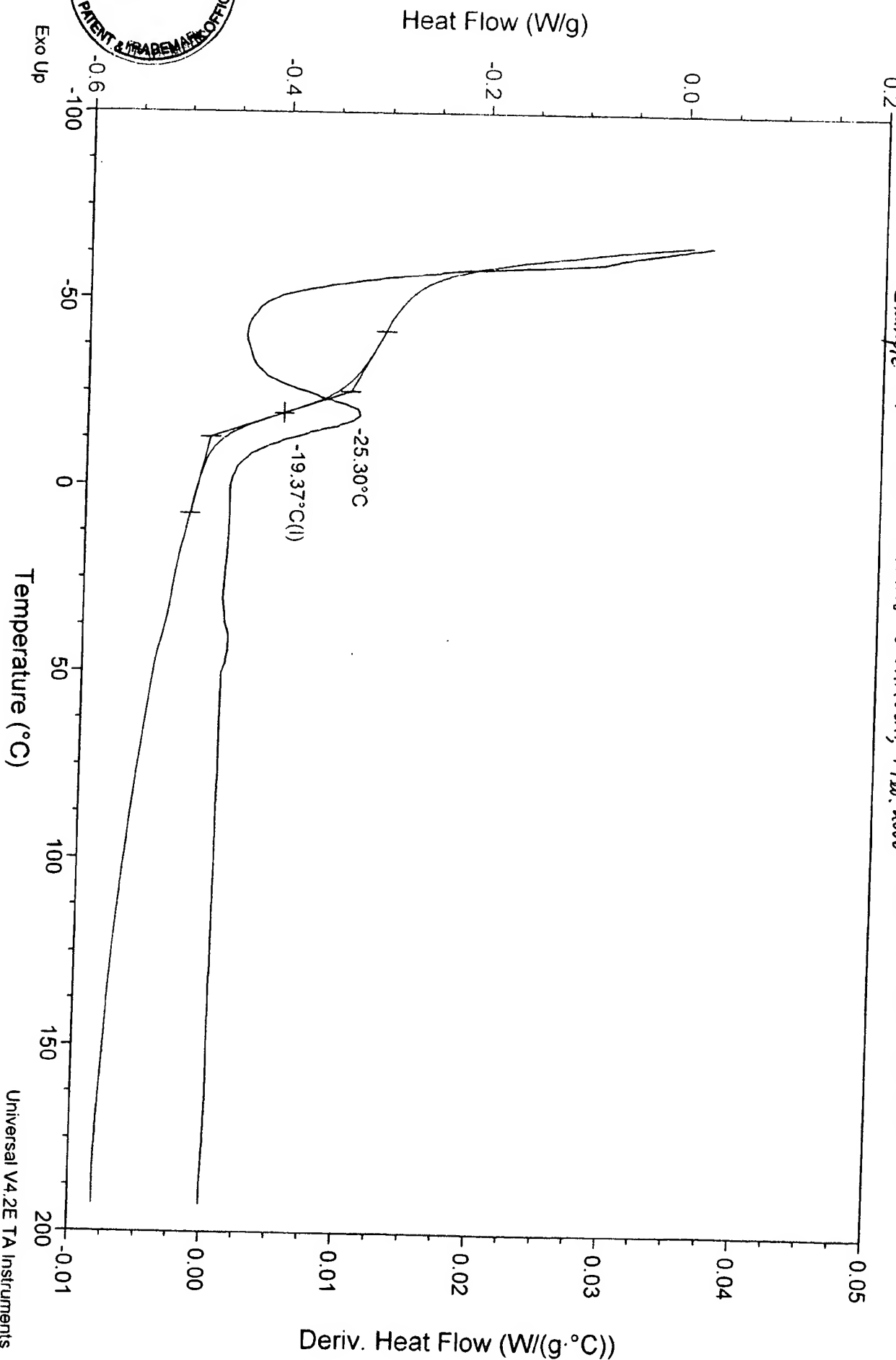
Date 7 February 2006

Sample: EA-113-R1  
Size: 31.6000 mg  
Method: TG(105)(-65)(200)

## DSC

Example #1 from Rubisco Declaration, 7 Feb. 2006

File: C:\TA\Data\DSC\coxegd\EA113R1.001  
Operator: J COXE  
Run Date: 01-Feb-2006 12:29  
Instrument: 2010 DSC V4.4E



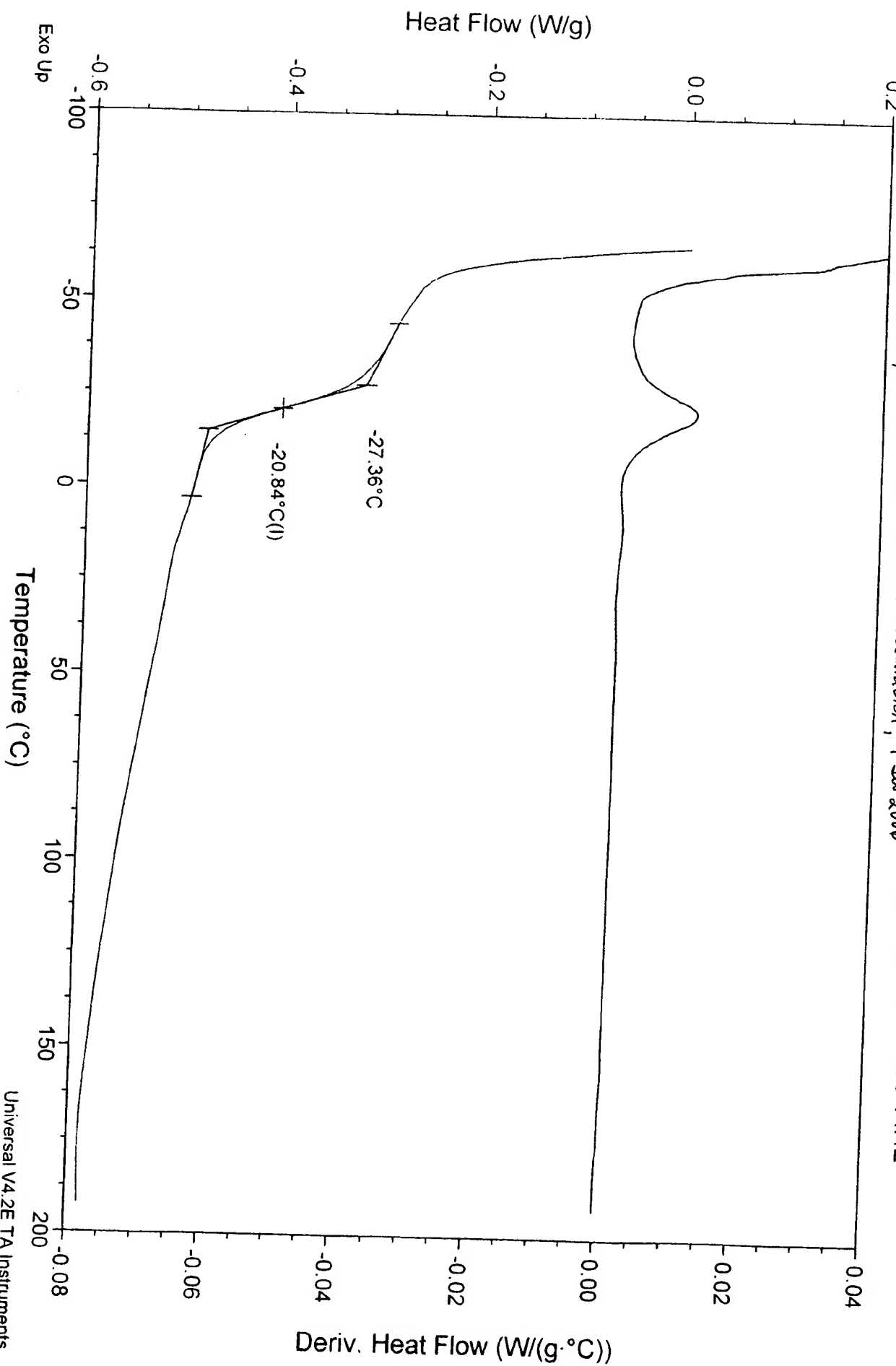


Sample: EA-114-R1  
Size: 29.7000 mg  
Method: TG(105)(-65)(200)

## DSC

Example #2 From Rabasco Declaration, 7 Feb 2006

File: C:\TA\Data\DSC\coxeqd\EA114R1.001  
Operator: J COXE  
Run Date: 19-Jan-2006 08:11  
Instrument: 2010 DSC V4.4E



Sample: AJ-661-R3  
Size: 17.0000 mg  
Method: TG(100)(-70)(200)  
Comment: ORION VAE JUR

# DSC

File: D:\DATA\COXEGD\AJ661R3  
Operator: J COXE  
Run Date: 20-Sep-02 07:51

Example #8 from case #06326 (10/6/20,654)

